



## Research article

# Cetyltrimethylammonium bromide intercalated and branched polyhydroxystyrene functionalized montmorillonite clay to sequester cationic dyes

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## ARTICLE INFO

## Keywords:

Cetyltrimethylammonium bromide  
Montmorillonite  
Rhodamine B  
Crystal violet  
Methylene blue  
Intercalation

## ABSTRACT

Herein, Cetyltrimethyl ammonium bromide (CTAB) intercalated and branched polyhydroxystyrene (BPS) functionalized montmorillonite (MMT) nano-composite (BPS-CTAB-MMT) was developed, characterized, and its potential as an adsorbent was tested in sequestering cationic dyes viz. rhodamine B (RB), crystal violet (CV), and methylene blue (MB) from aqueous environment.  $N_2$  adsorption/desorption isotherm showed mesoporous BPS-CTAB-MMT surface with a BET surface area of  $273.8 \text{ m}^2/\text{g}$ . The appearance of sharp spikes at  $2855$  and  $2925 \text{ cm}^{-1}$  (associated with symmetric and asymmetric tensions of C–H bonds) in infra-red spectrum of BPS-CTAB-MMT indicates successful intercalation of MMT with CTAB and functionalization with BPS. The observed crystallite size of BPS-CTAB-MMT was  $66 \text{ nm}$ . Comparatively greater weight loss for BPS-CTAB-MMT (11%) than MMT (9%) was observed during thermogravimetric analysis. The adsorption of dyes on BPS-CTAB-MMT was pH dependent with maximum uptake was observed in the pH range: 5–6. For initial dyes concentration ( $C_0$ ) range: 50–150 mg/L, the observed equilibration time for CV was 300 min, whereas for RB and MB the equilibration time varied between 300 and 360 min. Modeling investigations revealed the applicability of Sips isotherm and pseudo-second-order (PSO) kinetic models to dyes adsorption data. Sips maximum adsorption capacity ( $q_s$ ) values for RB, CV, and MB at  $55^\circ\text{C}$  were 476.5, 438.7, and 432.7 mg/g, respectively. The adsorption of dyes on BPS-CTAB-MMT was thermodynamically favorable. Desorption studies showed 42.1% RB and 41.9% CV recovery with 0.1 M NaOH and  $\text{CH}_3\text{COCH}_3$ , respectively, while only traces of MB were recovered with tested eluents.

## 1. Introduction

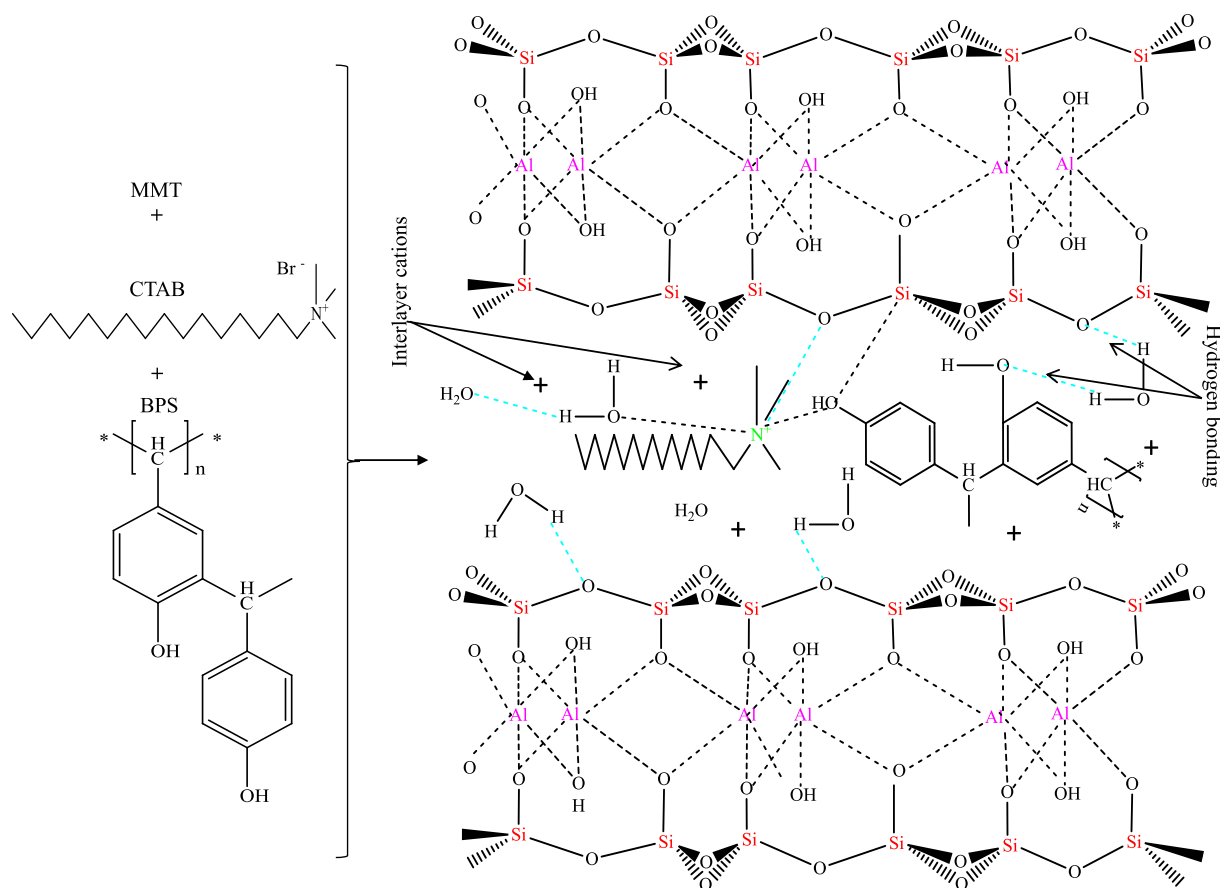
Dyes and pigments are widely used in textile, paper, plastic, rubber, concrete, paint, and pharmaceutical industries (Yusuf et al., 2017). Among them, textile industry is a major consumer of dyes. About 10% of the dyes used in industries are discharged into the surroundings posing potential environmental threats (Bazin et al., 2012). The dyes dissemination into the water bodies leads to colored water, which is a serious public concern. Also, the dispersed dyes molecules obstruct sunlight from reaching the bulk of the affected water system, leading to the decline in dissolved oxygen level in the polluted water. The presence of dyes may also increase the biochemical oxygen demand of the contaminated water (Ahmad et al., 2015). Dyes are non-biodegradable molecules. Most of the dyes are carcinogenic and often causes allergies

like dermatitis, and skin irritation (Yusuf et al., 2015). Due to their lethal impacts on biota, the treatment of dyes-contaminated effluent is essential.

Various treatment techniques and materials like adsorption, biological treatment, oxidation, ion-exchange, filtration, precipitation, electrolysis, reverse osmosis, coagulation, biofouling, and biodegradable materials have been employed to remove dyes from wastewater. Synthetic dyes, on the other hand cannot be efficiently decolorized by the conventional treatment methods viz. activated sludge process, coagulation, oxidation etc. Among the techniques stated above, adsorption is arguably the most sought after techniques for removing dyes from industrial effluents and wastewater owing to its excellent efficiency, process simplicity as well as cost effectiveness (Forgacs et al., 2004; Khan et al., 2008).

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Scheme 1. Intercalation and functionalization of MMT.

During recent years, the use of clay and clay minerals as an adsorbent and heterogeneous catalyst in water remediation operations has increased as they are cost-effective and ecologically safe. Montmorillonite (MMT), a di-octahedral layered cationic clay, classified under 2:1 smectite type clay minerals family, having a molecular formula  $M_x(Al_{2-x}Mg_x)(Si_4)O_{10}(OH)_2 \cdot nH_2O$  (Kumar et al., 2014). Structural arrangement of MMT showed the presence of an octahedral alumina sheet in between two tetrahedral silica sheets forming a layer. The distance between two consecutive aluminosilicate layers in MMT is known as interlayer spacing. The partial isomorphous substitution of ions present in between interlayer spacing of MMT causes charge imbalance. Therefore, to stabilize the charge deficit, hydrated alkali and alkaline earth metal cations occupy the interlayer spacing of MMT. These cations are highly exchangeable and thereby make MMT accommodative for various guest molecules in between its interlayer space.

Previous studies have reported the use of macromolecules such as surfactants in MMT intercalation and its applications in aqueous environment remediation. During intercalation process the interlayer spacing between the nearby MMT layers enlarged, and thus, increasing its surface area resulting in an enhanced sequestering of adsorbate ions. Due to an increase in interlayer gap, the surface energy reduces, resulting in conversion of hydrophilic silicate surface to hydrophobic and therefore, enhancing its adsorption potential (Liu et al., 2011). Wang and Wang (2008) observed a drastic increase in congo red adsorption on CTAB-MMT to 229 mg/g from 10.2 mg/g on MMT. Chen et al. (2011) reported the intercalation of MMT with both cationic (CTMAB) and anionic (SSTA) surfactants and determine its applicability in the removal of methyl orange. CTMAB/10SSTA-MMT showed the highest adsorption capacity for methyl orange. Gemini surfactants intercalated MMT was tested for the removal of methyl orange with an improved adsorption on (18-3-18)-MMT-3 (Liu et al., 2011). A highly effective

adsorption of acid red G was observed on octadecyltrimethyl ammonium-MMT (Tong et al., 2010). Dodecyl sulfobetaine surfactant-modified MMT showed an appreciably high adsorption capacity (254 mg/g) for methylene blue (Fan et al., 2014). Though, the previous studies have reported the intercalation of MMT by different surfactants and their applications in removing dyes from aqueous phase but comprehensive literature survey revealed that limited studies are available on functionalization of surfactant intercalated MMT.

In the present study, MMT was intercalated with cetyltrimethyl ammonium bromide (CTAB) to increase the interlayer spacing. Furthermore, to enhance its adsorption potential, CTAB-MMT was functionalized with branched polyhydroxystyrene (BPS). The developed CTAB-BPS-MMT nano-composite was characterized using XRD, FT-IR, DSC, SEM and TEM-EDX, TGA analysis and its application as an adsorbent was tested in the removal of cationic dyes viz., rhodamine B (RB), methylene blue (MB), crystal violet (CV) from aqueous medium. The effects of pH, temperature, initial dyes concentration, and contact time on the adsorption were studied. Isotherm, kinetics, and thermodynamic parameters were evaluated to calculate the dyes adsorption controlling mechanisms on intercalated CTAB-BPS-MMT nano-composite surface.

## 2. Experimental

### 2.1. Chemicals and reagents

Montmorillonite K10 (MMT) was procured from Aldrich, Germany. Cetyltrimethyl ammonium bromide (CTAB) was purchased from Sigma, USA. Crystal violet (CV), methylene blue (MB), and rhodamine B (RB) dyes were obtained from Merck and Sigma-Aldrich, Germany, respectively. Branched polyhydroxystyrene (BPS) was procured from

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